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Replacing GB/T 1509-2006

Manganese ores - Determination of silicon content - Perchloric acid dehydration gravimetric method

锰矿石 硅含量的测定 高氯酸脱水重量法

(ISO 5890:1981, Manganese ores and concentrates - Determination of silicon content - Gravimetric method, MOD)

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Manganese ores - Determination of silicon content - Perchloric acid dehydration gravimetric method

Warning - The personnel using this standard shall have practical experience in formal laboratory work. This standard does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This standard specifies the determination of silicon content in manganese ore by the perchloric acid dehydration gravimetric method.

This standard applies to the determination of silicon content in fluorine-free manganese ores and manganese concentrates; the measurement range (mass fraction): 0.50% to 20.00%.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 2011 Method of sampling and sample preparation of manganese ores in bulk (GB/T 2011-1987, neqISO3081:1983)

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

GB/T 14949.8 Manganese ores - Determination of hygroscopic moisture content in analytical samples (GB/T 14949.8-1994, eqv ISO 310:1981)

3 Principles

The sample is decomposed by hydrochloric acid and nitric acid, filtered; the residue is melted by sodium carbonate; the leaching liquid is combined with the

main liquid; perchloric acid is added to dehydrate the silicic acid; the precipitate is burnt, weighed, volatilized by hydrofluoric acid to remove the silicon. It is burnt and weighed. The mass difference before and after the treatment by hydrofluoric acid is used to calculate the silicon content.

4 Reagents and materials

In the analysis process, only use approved analytical reagents and water that meets the grade 3 or higher purity as specified in GB/T 6682.

4.1 Anhydrous sodium carbonate, solid.

4.2 Hydrochloric acid, ρ = 1.19 g/mL.

4.3 Hydrochloric acid, 1 + 4.

4.4 Hydrochloric acid, 1 + 9.

4.5 Nitric acid, ρ = 1.40 g/mL.

4.6 Sulfuric acid, 1 + 1.

4.7 Perchloric acid, ρ = 1.67 g/mL.

4.8 Hydrofluoric acid, ρ = 1.15 g/mL.

4.9 Hydrogen peroxide, ρ = 1.11 g/mL.

4.10 Ammonium thiocyanate solution, 50 g/L.

5 Instruments

Common laboratory instruments and equipment are used in the analysis.

6 Sampling and preparation

According to GB/T 2011, take sample and prepare it. The particle size of the sample shall not be greater than 0.080 mm.

7 Analytical procedures

7.1 Number of analyses

Perform at least two repeatability analyses on the same sample.

7.4.3 Separation of silicon

Add 30 mL of perchloric acid (see 4.7) to the solution obtained in 7.4.2. Heat and evaporate until the white smoke of perchloric acid begins to be emitted. Cover with a watch glass. Continue to heat until the perchloric acid is refluxed for 15 to 20 minutes. Remove it. Cool the solution. Add 40 mL ~ 50 mL of hot water, 2 ~ 3 drops of hydrogen peroxide (see 4.9). Add 5 mL of hydrochloric acid (see 4.2). Heat to dissolve soluble salts until the solution is clear. Immediately use medium-speed quantitative filter paper with a little pulp to filter it. Use a glass rod with a rubber tip to transfer the silicon precipitate in the beaker to the filter paper.

First use cold water to wash the beaker and the precipitate 2 ~ 3 times. Then use hot hydrochloric acid (see 4.4) to wash it, until there is no iron ions (test with ammonium thiocyanate solution (see 4.10), if the ammonium thiocyanate solution turns red, it indicates that there is still iron ion]. Finally use cold water to wash it 2 ~ 3 times. For samples with silicon content (mass fraction) below 10%, discard the filtrate and washing liquid. For samples with a silicon content (mass fraction) of more than 10%, add 20 mL of perchloric acid (see 4.7) to the filtrate. Repeat evaporation and smoking, filtration, washing; the resulting precipitate is processed according to operation step 7.4.4.

Note 1: The surface of the solution is calm when heating and evaporating to the beginning of white smoke of perchloric acid.

Note 2: There is a downward flow on the cup wall during the reflux of the perchloric acid smoke; the solution gradually forms a gel.

7.4.4 Treatment of silicon precipitate

Put the precipitate together with the filter paper in a platinum crucible. At low temperature, heat to dry and carbonize it. Place it in a high temperature furnace at 700 °C ~ 750 °C for ashing. Then increase the temperature to 950 °C ~ 1000 °C. Burn to a constant amount. Take it out. Place it in a desiccator. Cool to room temperature. Weigh the mass of the crucible and the precipitate (m_1).

Use a few drops of water to wet the precipitate in the platinum crucible. Add 5 ~ 10 drops of sulfuric acid (see 4.6), 5 mL to 10 mL of hydrofluoric acid (see 4.8). Slowly evaporate on an electric furnace until silicon and sulfuric acid are completely volatilized. Place the platinum crucible containing the residue in a high temperature furnace at 950 °C ~ 1000 °C and burn to a constant amount. Take it out. Place it in a desiccator. Cool to room temperature. Weigh the crucible and the mass of the residue (m_2).

Note: When silicon and sulfuric acid are completely volatilized, there will be no more smoke in the platinum crucible.

8.2.2 Acceptance of analytical values of standard samples

When analyzing the sample, it is necessary to analyze the same type of standard sample along with the sample. The analytical value of the standard sample shall meet the conditions of formula (2):

$$|y-\mu_0| \leqslant k_3 R \qquad \cdots\cdots(2)$$

Where:

y - The analytical value of the standard sample;

μ_0 - The definite standard value of the standard sample;

k_3 - 0.7, the critical difference coefficient;

R - Allowable difference.

If the analytical value of the standard sample does not conform to formula (2), it needs to be re-analyzed together with the sample.

If the two analytical values of the sample exceed the allowable error limit given in Table 2, one or more additional analyses shall be performed simultaneously with the standard sample of the same type of ore according to the flowchart in Appendix A.

In any case, the acceptability of the analytical value of the sample shall depend on the acceptability of the analytical value of the standard sample.

8.2.3 Calculation of final result

The final result is the arithmetic average of the acceptable value of the sample, or the analytical value of the sample obtained according to the process specified in Appendix A. The value is rounded off according to the provisions of GB/T 8170, retaining two decimal places.

9 Test report

The test report shall include the following:

a) The name and address of the testing laboratory;

b) Date of release of test report;

c) The number of this standard;

d) The necessary detailed description of the sample itself;

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